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GAS TURBINE AND JET ENGINE FUELS

PROGRESS REPORT NO. 6

NAVY CONTRACT N600(19)-58219

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PHILLIPS PETROLEUM COMPANY

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By

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S U M M A R Y

Twelve-hour metal loss tests on the effect of sulfur on turbine inlet guide vanes using the Phillips 2-Inch Research Combustor operating under conditions producing 2000 F exhaust gases have been completed during the sixth bimonthly period, February through March, 1963, under Navy Contract N600(19)-58219. Data are now available on sulfur-free and 1.0 per cent sulfur fuels. Five typical current-generation superalloys including Udimet 500, Waspalloy, Haynes Alloy 25, Rene 41 and Hastelloy R-235 have been evaluated. Changes in tensile strength as a result of exposure to these atmospheres have also been determined. Some time has also been spent during this period on exploratory "black plague" corrosion tests (discussed in Progress Report No. 5) attempting to operate the current 2-inch combustor under cyclic conditions simulating a low-level supersonic tactical fighter mission.

Results of the 12-hour guide vane metal loss tests indicate that 1 per cent fuel sulfur or, rather, the gaseous sulfur compounds produced upon combustion of this sulfur, had no effect upon the durability of any of the alloys tested. Haynes Alloy 25, a cobalt-base alloy, showed best performance, Hastelloy R-235 poorest performance. These extremes differed by a factor of about five. The balance of the alloys performed comparably and intermediate between these extremes. Only Haynes Alloy 25 and Waspalloy showed more degradation of tensile strength when exposed to a sulfur atmosphere than when exposed to a sulfur-free atmosphere. HA25 lost nearly twice as much tensile strength with sulfur as without. The indicated degradation of Waspalloy strength by sulfur is considered only marginally significant. All the alloys suffered marked losses in ductility after exposure, suggesting the occurrence of some intergranular oxidation.

The exploratory "black plague" tests conducted during this reporting period have shown that the present 2-inch combustor design is not satisfactory for operation under conditions simulating a Mach 2+ low level tactical fighter mission because of repeated liner failures by melting. Design and development of new combustors to satisfy these requirements is in progress.

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PHILLIPS PETROLEUM COMPANY

BARTLESVILLE, OKLAHOMA

Progress Report No. 6
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I. INTRODUCTION

Twelve-hour metal loss tests on the effect of sulfur on turbine inlet guide vanes using the Phillips 2-Inch Research Combustor operating under conditions producing 2000 F exhaust gases have been completed during the sixth bimonthly period, February through March, 1963, under Navy Contract N600(19)-58219. Five typical current generation superalloys including Udimet 500, Waspalloy, Haynes Alloy 25, Hastelloy R-235 and René 41 have been evaluated. An essentially sulfur-free (<10 ppm total sulfur) JP-5 boiling range isoparaffinic fuel was used as the base fuel and was artificially contaminated with 1.0 per cent sulfur, as ditertiary butyl disulfide. The tensile strengths of the alloy specimens were also evaluated following exposure in this high temperature sulfurous atmosphere. The results of these tests, when compared with those on the sulfur-free fuel reported in Progress Report No. 3, provide a direct expression of the effects of fuel sulfur contamination on the durability of these superalloys at 2000 F.

In addition to the test work mentioned above, some time has been spent during this reporting period on some exploratory testing with the objective of establishing a suitable procedure and combustor operating conditions for evaluating sulfur corrosion of the type known colloquially as "black plague". A discussion of this phenomenon has previously been presented in Progress Report No. 5. The results of this preliminary testing will also be discussed herein.

II. TEST METHODS AND APPARATUS

The Phillips 2-Inch Research Combustor and associated equipment used in preparing the combustion-gas-exposed simulated turbine inlet guide vane specimens have been described in previous reports (1, 2, 3). This combustor was operated under conditions simulating sea level takeoff and low altitude cruise ($P = 350$ in Hg absolute, $V = 100$ ft/sec, $IAT = 700$ F, $F/A = 0.020$) of a 12:1 compression ratio turbojet. These conditions resulted in an exhaust gas temperature (turbine inlet temperature) of approximately 2000 F. Simulated turbine inlet guide vanes were placed, as shown in Figure 1, at a position approximately six inches downstream from the combustor section. The duration of exposure of the pair of specimens was twelve hours, obtained by running six two-hour intervals, the specimens being descaled by rotary wire brushing at the end of each two-hour period. The data are reported as milligrams of accumulated metal loss (both strips) per square centimeter of exposed area. Data have previously been reported (3) on metal durability during exposure to combustion gases derived from the (essentially) sulfur-free base fuel and are included herein for comparison with present data on the base fuel contaminated with 1.0 per cent sulfur (as ditertiary butyl disulfide).

The exposed specimens of each alloy were fabricated into the shape shown in Figure 2 and their tensile strengths determined using an Instron Model TCC testing machine operated at a crosshead travel of 0.1 inch per minute. Gage length was one inch. Tensile data on unexposed samples and on those exposed for twelve-hours with the sulfur-free base fuel have previously been reported (2, 4). Table I shows the compositions of the alloys evaluated.

III. DISCUSSION OF EXPERIMENTAL RESULTS

The data obtained during this period on metal loss from Waspalloy, Udimet 500, Haynes Alloy 25, René 41 and Hastelloy R-235 simulated turbine inlet guide vanes are shown in Table II and Figures 3 and 4. Data previously obtained on the same base fuel without sulfur are also shown for purposes of comparison. All of these tests were run at a nominal exhaust gas temperature of 2000 F, which is representative of maximum turbine inlet temperature in advanced design engines. With the exception of Udimet 500, these 12-hour extended tests were conducted using the original test specimens from the six-hour tests reported in (2). This was also attempted with the original Udimet 500 strips but malfunction of the propane igniter system caused flame impingement on these specimens resulting in their destruction. Therefore, it was necessary to restart the Udimet 500 test with new strips.

Figure 3 indicates that no marked acceleration of metal loss rate was observed between six and twelve hours either with or without sulfur in the fuel. In addition, extension of the test duration did not alter the relative rankings of the alloys appreciably.

It will be observed from Figure 4 that 1.0 per cent sulfur in the isoparaaffinic base fuel had either no effect or an apparent beneficial effect on the durability of this group of superalloys. A statistical analysis of variance was performed on these data. This analysis resulted in acceptance of the null hypothesis; that is, that there is no difference between the means of the two fuels (base and base plus 1 per cent sulfur). These tests suggest that 1.0 per cent sulfur is neither beneficial nor detrimental from the standpoint of turbine inlet guide vane metal loss. However, the analysis of these data in terms of alloy effects resulted in a rejection of the null hypothesis; that is, a significant difference among means of the different metals is indicated. The results of the analysis of variance are shown in Appendix I.

Considering the results shown in Figure 4 in terms of the relative durability of the several alloys, it will be noted that the extremes are Haynes Alloy 25 (lowest loss) and Hastelloy R-235 (highest loss). These extremes differ by a factor of about five while the balance of the alloys lie intermediate and differ from each other only by values believed to lie within the repeatability of the test method and apparatus.

The data obtained during this period on tensile strengths after 12-hours exposure in a sulfurous atmosphere are shown in Table III and Figures 5, 6, 7, 8, and 9 together with previous data before exposure (new) and after 12-hours exposure using the sulfur-free base fuel. As indicated in Table III only two of the alloys, Waspalloy and Haynes Alloy 25 showed more degradation of tensile strength by the addition of 1.0 per cent sulfur to the fuel than was observed with the sulfur-free fuel. Of these two materials, the effect of sulfur was by far the greatest with Haynes Alloy 25, this material losing nearly twice as much strength with sulfur as without. The degradation of Waspalloy by sulfur is much less definite but is, at least, indicated. Calculation of the least significant difference (LSD) between a pair of observations (95 per cent confidence), when based upon the pooled standard deviation observed for the duplicate tests available on the unexposed samples, results in a value of 5750 psi. Thus, a pair of tensile strength values would have to differ by 5750 psi or more before one could justifiably conclude that a real difference exists. Consequently, the strength changes shown in Table III for Waspalloy and Hastelloy R-235 exposed to base fuel only and for Hastelloy R-235 exposed to base fuel plus 1.0 per cent sulfur are not believed significant.

With the exception of the tensile data on Haynes Alloy 25, which differs from the balance of the alloys in that it is a cobalt base alloy rather than nickel base, these results do not provide a condemnation of fuel sulfur

contamination from the standpoint of effects on component strength. Rather, they suggest a null effect of sulfur on tensile strength for the four nickel base alloys.

In addition to changes in tensile strength it will be observed from Figures 5, 6, 7, 8, and 9 that all of the alloys suffered marked losses in ductility after 12 hours exposure in the combustor with and without sulfur, as shown by the fact that all specimens failed at much lower strains than before exposure. In general, these losses were no greater with sulfur than without. Of additional interest is the fact that, in general, the "after exposure" curves followed the "before exposure" curves within the elastic range. One explanation of the decrease in ductility with loss in strength may lie in the possibility of intergranular oxidation during exposure in the combustor. These oxides might bestow little overall change in strength during elastic deformation, but be unable to withstand plastic deformation, thus resulting in failure at both lower strains and lower stresses. Several of the photomicrographs currently available on some of the exposed specimens do show evidence of intergranular oxidation, lending credence to this explanation.

IV. EXPLORATORY "BLACK PLAGUE" CORROSION TESTS IN 2-INCH COMBUSTOR

Following completion of the tests described above some exploratory "black plague" turbine inlet guide vane corrosion tests were attempted with the two-inch combustor operating on a 1.0 per cent sulfur fuel containing 25 per cent (wt) aromatics. Sea water was injected, thus completing the environment of sulfur dioxide, sea salt, and reducing carbon by virtue of maximum specification aromatics content in the fuel. These tests were run under cyclic conditions simulating a low-level tactical fighter mission consisting of 600 miles at Mach 1.2 and 200 miles at Mach 2.2. Specifically, the combustor operating conditions chosen for these tests were as follows:

Cycle	Flight Conditions Simulated	2-Inch Combustor Operating Conditions				
		P, in Hg Abs	V Ft/Sec	Inlet Air Temp, F	Exh. Gas Temp., F	Duration, Minutes
1	300 Miles Sea Level Cruise to Target Area at Mach 1.2	350	200	900	1550-1600	20
2	200 Miles Sea Level Super- sonic Dash Over Target Area at Mach 2.2	450	200	1200	1950-2000	10
3	300 Miles Sea Level Cruise Return to Base at Mach 1.2	350	200	900	1550-1600	20
4	Deceleration, Landing and Shutdown	350	200	900	900	5
		350	100	900	900	5

The intention was to repeat this sequence six times for a total test duration of six hours or six simulated missions. Attempts to operate the present Phillips 2-Inch Research Combustor under the very severe conditions of Cycle 2 resulted in melting and subsequent catastrophic failures of combustor liners. Various expedients, such as adjustment of cooling air port sizes, were tried without success. This was followed by several tests at a compromise inlet air temperature of 1100 F in an attempt to alleviate the obvious cooling problem. These tests produced varying results ranging from failure at the end of the first complete sequence to failure at the end of five sequences. Thus, although some improvement did accrue, liner performance was not sufficiently dependable.

At this point it became clear that the present combustor design was not satisfactory for operation under the above conditions. Further attempts to operate under these conditions were suspended and combustor redesign studies were initiated. Early evaluations of a first redesigned combustor have yielded promising, although not yet completely successful, results.

In the meantime, in order to produce data on "black plague" corrosion prior to the termination of the present contract, a series of tests has been started operating under compromised combustor operating conditions of $P = 350$

in Hg absolute, $V = 100$ ft/sec, IAT = 900 F and EGT = 1950-2000 F. Again, the base fuel contains 25 per cent aromatics and is doped with 1.0 per cent sulfur. Synthetic sea water is also being injected during these runs. This testing is currently in progress and will hopefully be completed by the end of the current contract period, May 31, 1963.

V. CONCLUSIONS

Twelve-hour two-inch combustor tests completed during the present reporting period on the effects of sulfur on the loss of metal from simulated turbine inlet guide vanes operating at 2000 F have shown the following:

1. Guide vane metal loss is approximately a linear function of exposure time regardless of fuel sulfur content. No marked acceleration of metal loss rate was observed between six and twelve hours for the five superalloys tested.
2. Extension of test duration from six to twelve hours did not alter the relative rankings of the five superalloys appreciably. Haynes Alloy 25 remained lowest in metal loss, Hastelloy R-235 highest. Udimet 500, Waspalloy and René 41 were comparable and intermediate between these extremes.
3. Conventional evaluation of the data indicates that the addition of 1.0 per cent sulfur to the isoparaffinic base fuel had either no effect or an apparent beneficial effect on the durability of this group of alloys. Analysis by statistical methods indicates that the fuel sulfur had no effect on metal durability.

Tensile strength tests conducted on simulated turbine inlet guide vane specimens before and after twelve hours exposure to sulfur-free and sulfurous exhaust gases at 2000 F in the Phillips 2-Inch Research Combustor have shown the following:

1. Only two of the five alloys tested, Haynes Alloy 25 and Waspalloy, showed more degradation of tensile strength by the addition of 1.0 per cent sulfur to the fuel than was observed with the sulfur-free fuel. Haynes Alloy 25 lost nearly twice as much tensile strength with sulfur as without. The indicated degradation of Waspalloy by sulfur is considered only marginally significant.
2. All alloys tested suffered marked losses in ductility following exposure in the combustor. No sulfur effect was indicated.
3. In general, the "after exposure" stress-strain curves followed the "before exposure" curves within the elastic range. This suggests the formation of intergranular oxides which might alter the strength very little during elastic deformation, but be unable to withstand plastic deformation thus resulting in failure at both lower strains and lower stresses.

VI. OUTLINE OF PROJECTED EFFORTS

"Black plague" corrosion tests currently in progress using the current 2-inch combustor design operating with a fuel containing maximum specification limit aromatics content plus 1.0 per cent sulfur and injecting synthetic sea water will be continued during April and May. Further developmental testing of new combustor designs is also planned in order that future operations can be conducted with inlet air temperatures at or near 1200 F.

REFERENCES

1. Fromm, E. H.; "Design and Calibration of Phillips Jet Fuel Testing Facilities", Phillips Research Division Report 1252-55R, December, 1955.
2. Streets, William L.; "Gas Turbine and Jet Engine Fuels", Progress Report No. 2, Navy Contract N600(19)-58219, Phillips Research Division Report 3273-62R, August, 1962.
3. Streets, William L. ; "Gas Turbine and Jet Engine Fuels", Progress Report No. 3, Navy Contract N600(19)-58219, Phillips Research Division Report 3329-62R, October, 1962.
4. Streets, W. L.; "Gas Turbine and Jet Engine Fuels", Progress Report No. 4, Navy Contract N600(19)-58219, Phillips Research Division Report 3375-62R, November, 1962.

TABLE I
COMPOSITION OF ALLOYS USED IN TURBINE GUIDE VANE METAL LOSS TESTS

Alloy	Per Cent by Weight of Indicated Metal																
	Cr	W	Fe	C	Si	Co	Ni	Mn	Cb	Mo	P	S	Al	Ti	B	Zr	Cu
Udimet 500*	19.0	-	.36	.09	.15	18.7	51.04	<.10	--	4.35	-	.005	3.10	2.99	.003	<.01	<.10
Waspalloy*	19.5	-	.95	.053	.03	13.2	57.44	.01	--	4.41	.003	.003	1.23	3.10	.002	.06	.01
Haynes Alloy 25	20.0	15.0	3.0	.1	1.0	Bal.	10.0	1.5	--	-	-	-	-	-	-	-	-
Hastelloy R-235*	15.29	-	9.96	.15	.26	.38	63.91	.03	--	5.48	.001	.009	2.05	2.48	-	-	-
René 41*	18.33	-	1.90	.10	.16	10.69	54.37	.05	--	9.69	-	.009	1.54	3.15	.005	-	-

* Specific analyses for particular samples tested in combustor. Values shown for other alloys are typical compositions.

TABLE II
TURBINE GUIDE VANE DURABILITY TESTS IN PHILLIPS 2-INCH RESEARCH COMBUSTOR

Combustor Operating Conditions: P = 350 in Hg Abs; V = 100 ft/sec; IAT = 700 F; F/A = 0.020; EGT = 2000 F Nominal

Guide Vane Alloy	Test Fuel	Accumulated Metal Loss, mg/cm ²					
		2 Hr.	4 Hr.	6 Hr.	8 Hr.	10 Hr.	12 Hr.
Udimet 500	JP-5 Type Isoparaffinic Base Fuel	0.2	1.6	3.3	5.1	7.3	9.9
	Base Fuel + 1 % (Wt) Sulfur*	0.5	1.7	3.6	4.5	5.9	7.6
Waspalloy	JP-5 Type Isoparaffinic Base Fuel	2.8	3.9	5.8	7.6	10.1	12.6
	Base Fuel + 1 % (Wt) Sulfur*	1.5	3.4	4.7	7.1	9.1	12.9
René 41	JP-5 Type Isoparaffinic Base Fuel	1.2	2.7	4.4	6.8	9.2	12.7
	Base Fuel + 1 % (Wt) Sulfur*	1.5	3.0	5.1	6.1	7.7	9.7
Haynes Alloy 25	JP-5 Type Isoparaffinic Base Fuel	0.2	0.6	1.7	1.8	3.0	4.8
	Base Fuel + 1 % (Wt) Sulfur*	0.1	1.0	1.7	2.0	2.6	3.2
Hastelloy R-235	JP-5 Type Isoparaffinic Base Fuel	2.0	5.5	7.7	10.7	13.2	18.1
	Base Fuel + 1 % (Wt) Sulfur*	0.9	4.3	6.9	9.6	12.3	14.8

* Sulfur added as ditertiary butyl disulfide.

TABLE III
EFFECT OF FUEL SULFUR CONTAMINATION ON TENSILE STRENGTH OF SUPERALLOYS FOLLOWING
TWELVE HOURS EXPOSURE AT 2000 F IN THE PHILLIPS 2-INCH RESEARCH COMBUSTOR

Alloy	Tensile Strength Before Exposure, psi	Tensile Strength After 12 Hour Exposure @ 2000F w/Base Fuel, psi	Tensile Strength After 12 Hour Exposure @ 2000F w/Base Fuel + 1 % Sulfur, psi	Change in Tensile Strength After Exposure to Sulfur-Free Exhaust Gases, psi	Change in Tensile Strength After Exposure to Sulfurous Exhaust Gases, psi
Udimet 500	156,380	125,425	133,930	- 30,955	- 22,450
Waspalloy	120,650	124,415	111,530	+ 3,765*	- 9,120
René 41	136,000	119,400	120,760	- 16,600	- 15,240
Haynes Alloy 25	146,750	126,670	107,470	- 20,080	- 39,280
Hastelloy R-235	130,300	125,470	133,090	- 4,830*	+ 2,790*

* These changes are insignificant since they are less than the least significant difference (95 per cent confidence) of 5750 psi calculated from data on the unexposed specimens.

APPENDIX I

ANALYSIS OF VARIANCE (TWO VARIABLES OF CLASSIFICATION - SINGLE
OBSERVATIONS) OF TWO INCH COMBUSTOR DATA ON DURABILITY OF
SUPERALLOY TURBINE GUIDE VANES

	<u>Sum of Squares</u>	<u>Degrees of Freedom</u>	<u>Mean Square</u>
Metal Means	172.366	4	43.0915
Fuel Means	9.801	1	9.8010
Residual	4.114	4	1.0285
	<hr/>	<hr/>	<hr/>
Total	186.281	9	

$$F (\text{Metal means}) = \frac{43.0915}{1.0285} = 41.90 ; F_{.975} (4,4) = 9.60$$

Reject hypothesis of no difference among means of different metals.

$$F (\text{Fuel means}) = \frac{9.8010}{1.0285} = 9.53 ; F_{.975} (1,4) = 12.20$$

Accept hypothesis of no difference among means of different fuels.

HOLDER MAT'L: 310 SS; STRIP MAT'L.: VARIOUS TURBINE BLADING ALLOYS
SCALE: 1" = 1"

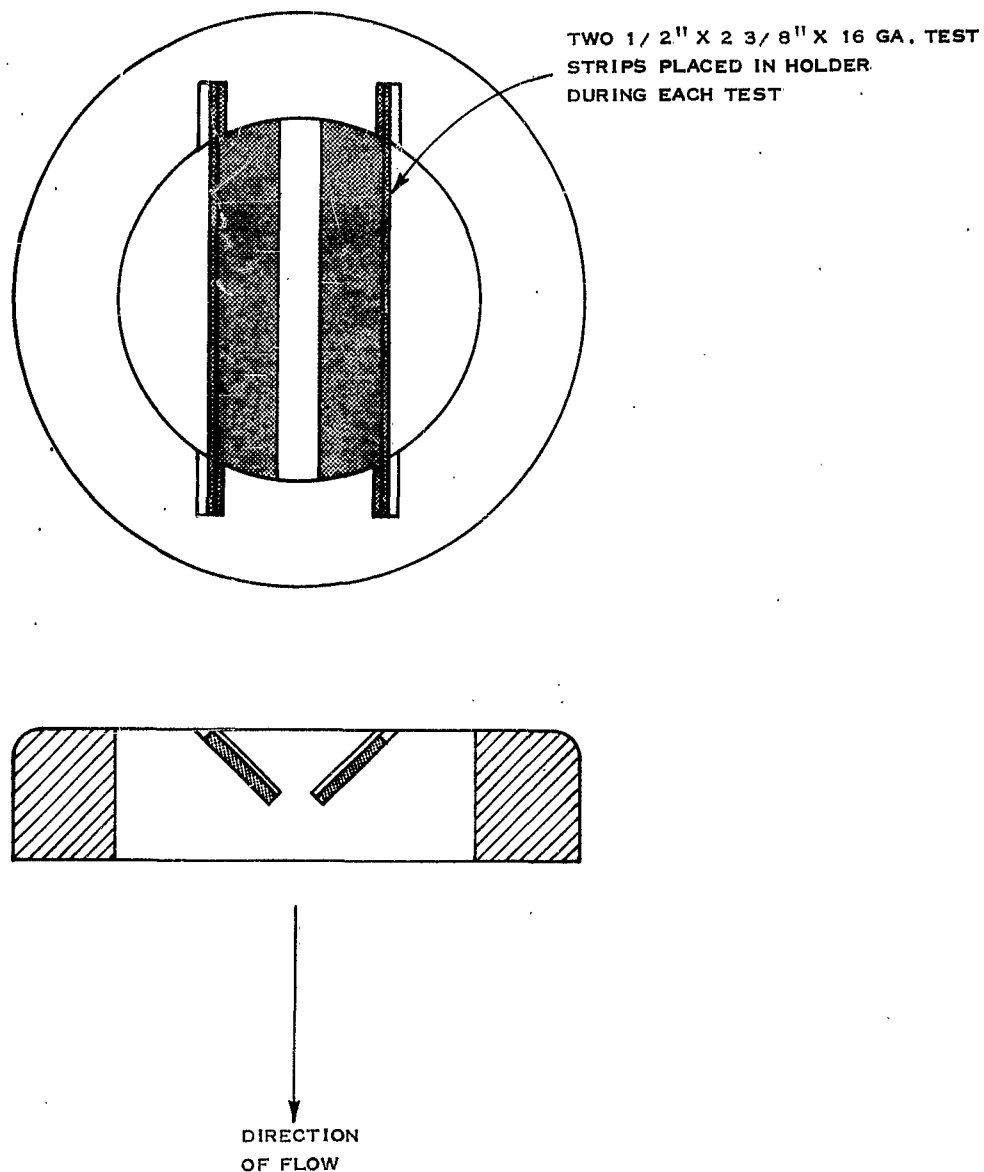
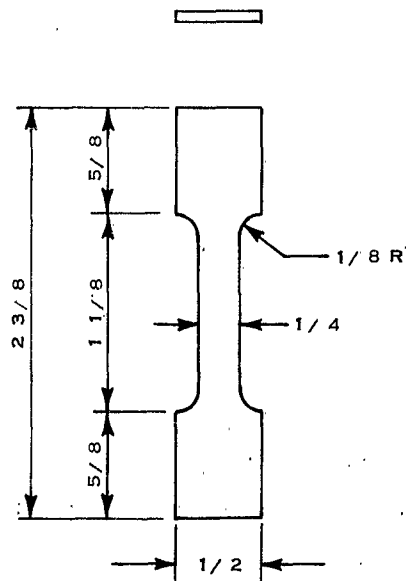


FIGURE 1
SPECIMEN HOLDER FOR PHILLIPS 2-INCH COMBUSTOR SIMULATED
TURBINE INLET GUIDE VANE DURABILITY TESTS



NOTE: TENSILE SPECIMENS TO BE FABRICATED FROM $\frac{1}{2} \times 2 \frac{3}{8} \times 16$ GAGE
NEW AND EXPOSED CORROSION TEST STRIPS FROM 2-INCH COMBUSTOR TESTS.

FIGURE 2
TENSILE TEST CONFIGURATION FOR TURBINE INLET GUIDE VANE
CORROSION SPECIMENS

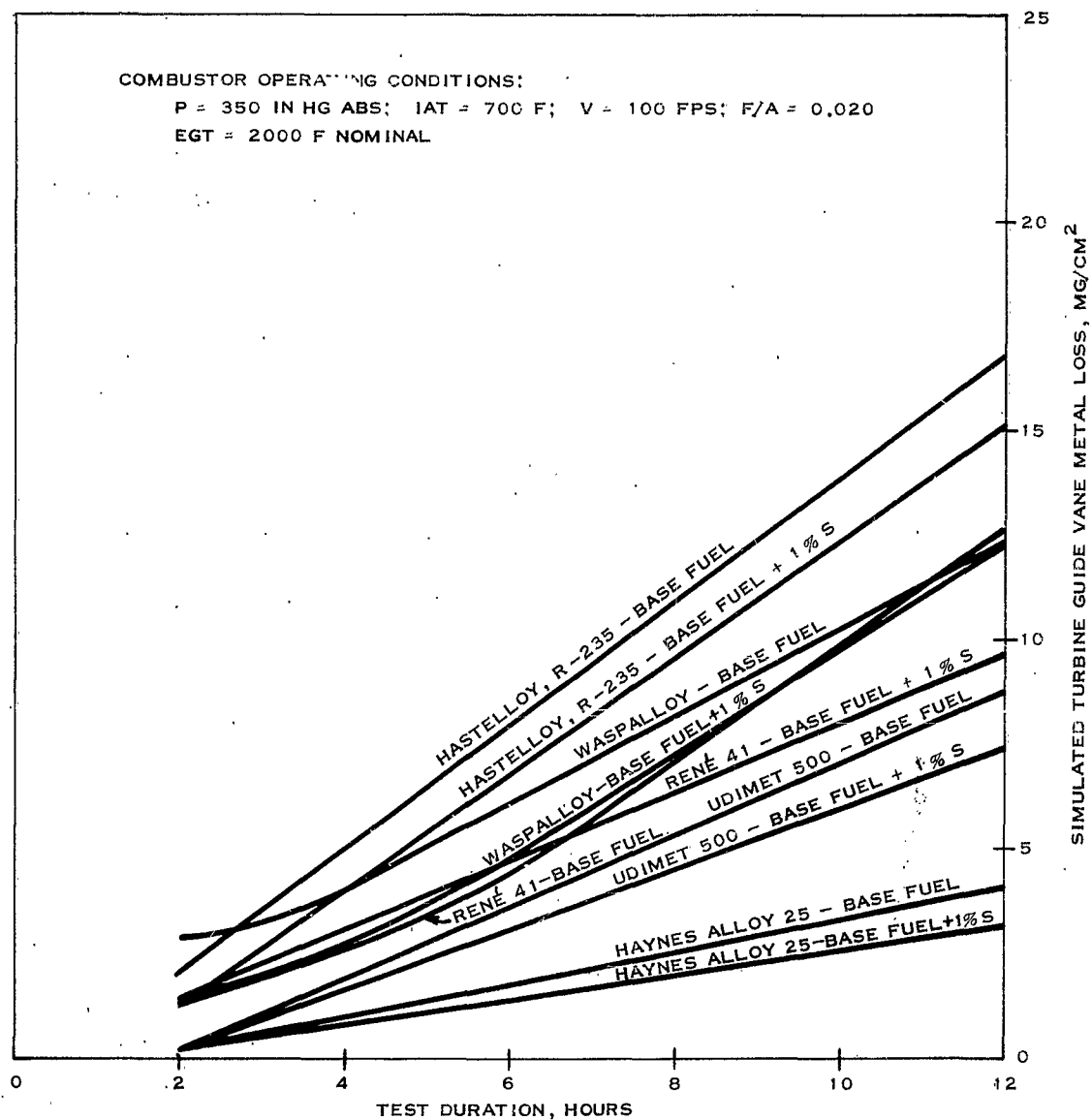


FIGURE 3
TURBINE GUIDE VANE METAL LOSS AS A FUNCTION OF TEST DURATION

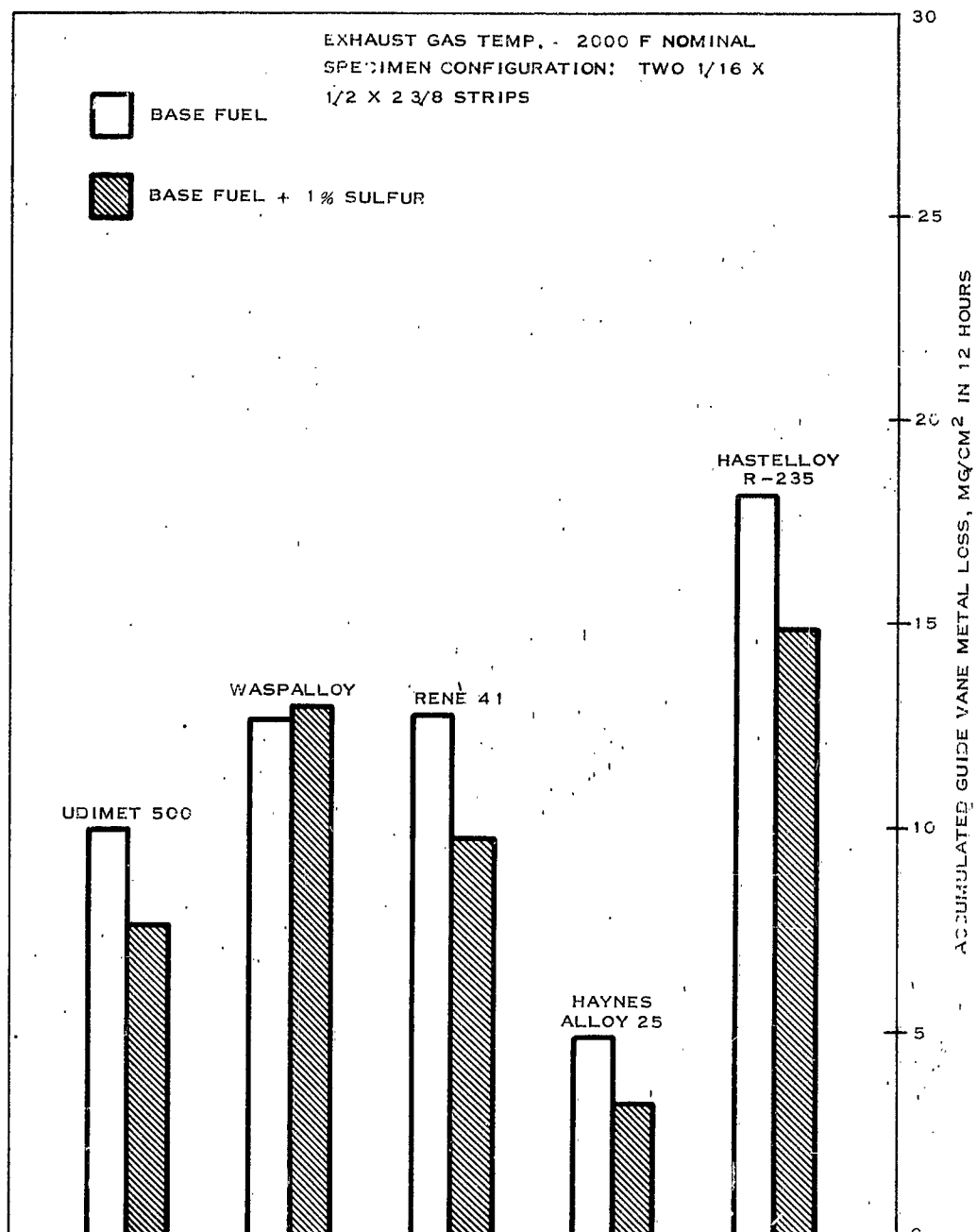


FIGURE 4
EFFECT OF FUEL SULFUR CONTAMINATION ON TURBINE GUIDE
VANE METAL LOSS FROM SEVERAL SUPERALLOYS IN THE PHILLIPS 2-INCH COMBUSTOR

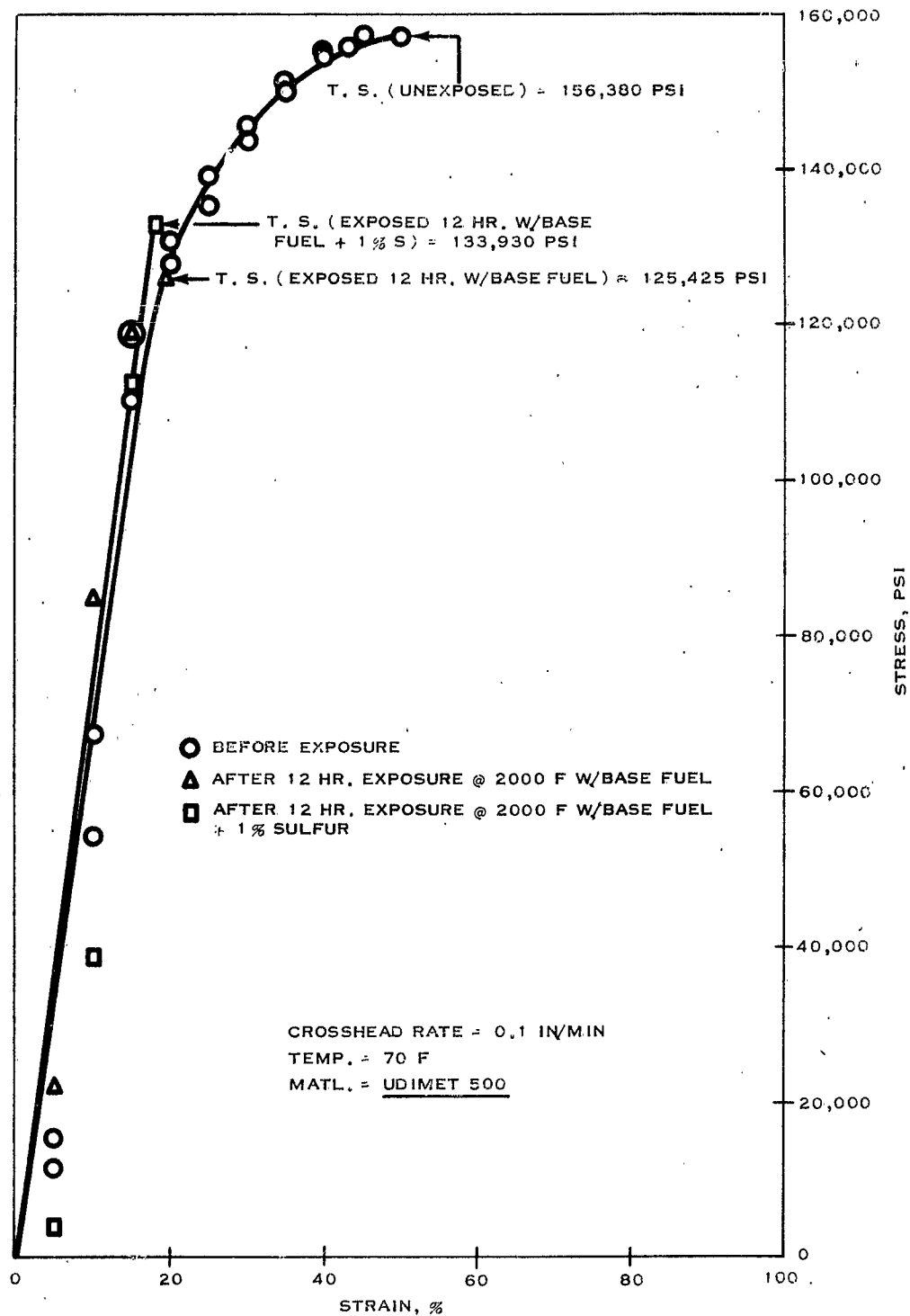


FIGURE 5
 STRESS-STRAIN CURVE FOR UDIMET 500 BEFORE AND AFTER
 12 HOURS EXPOSURE TO SULFUR-FREE AND SULFUROUS EXHAUST GAS @ 2000 F
 IN THE PHILLIPS 2-INCH RESEARCH COMBUSTOR

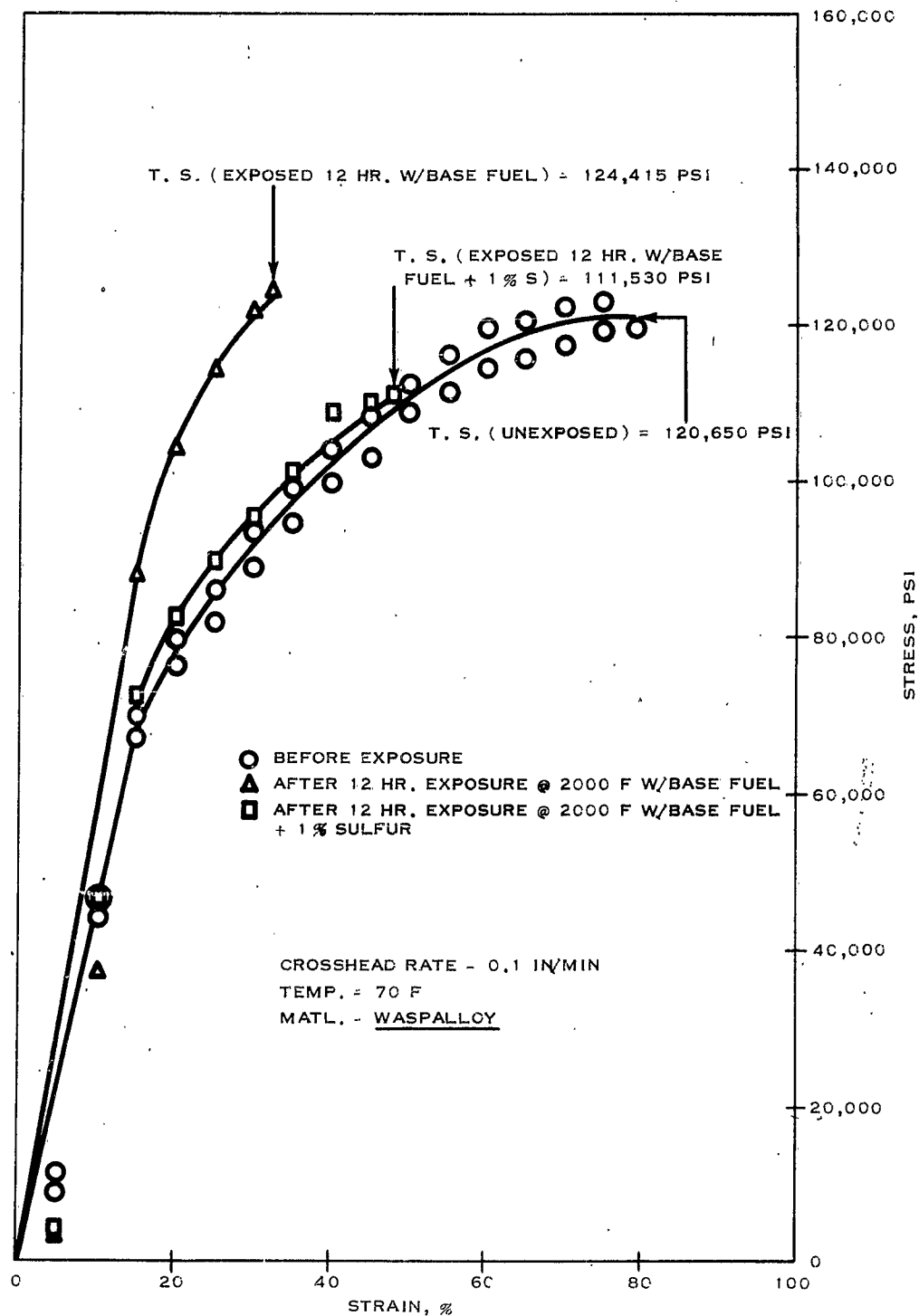


FIGURE 6
STRESS-STRAIN CURVE FOR WASPALLOY BEFORE AND AFTER 12 HOURS
EXPOSURE TO SULFUR-FREE AND SULFUROUS EXHAUST GAS @ 2000 F
IN THE PHILLIPS 2-INCH RESEARCH COMBUSTOR

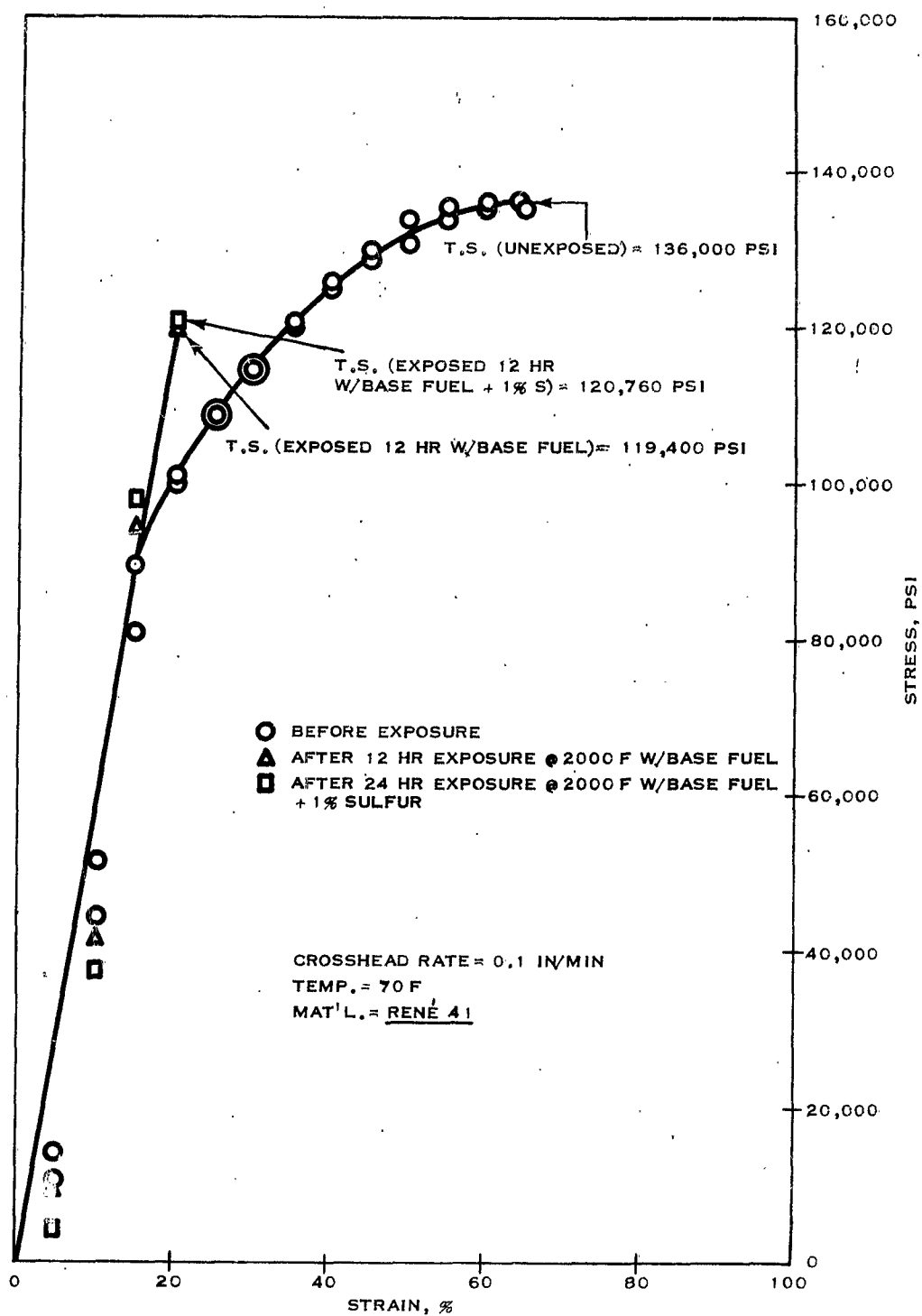


FIGURE 7
STRESS- STRAIN CURVE FOR RENE 41 BEFORE AND AFTER 12 HOURS
EXPOSURE TO SULFUR- FREE AND SULFUROUS EXHAUST GAS @ 2000 F IN
THE PHILLIPS 2- INCH REASEARCH COMBUSTOR

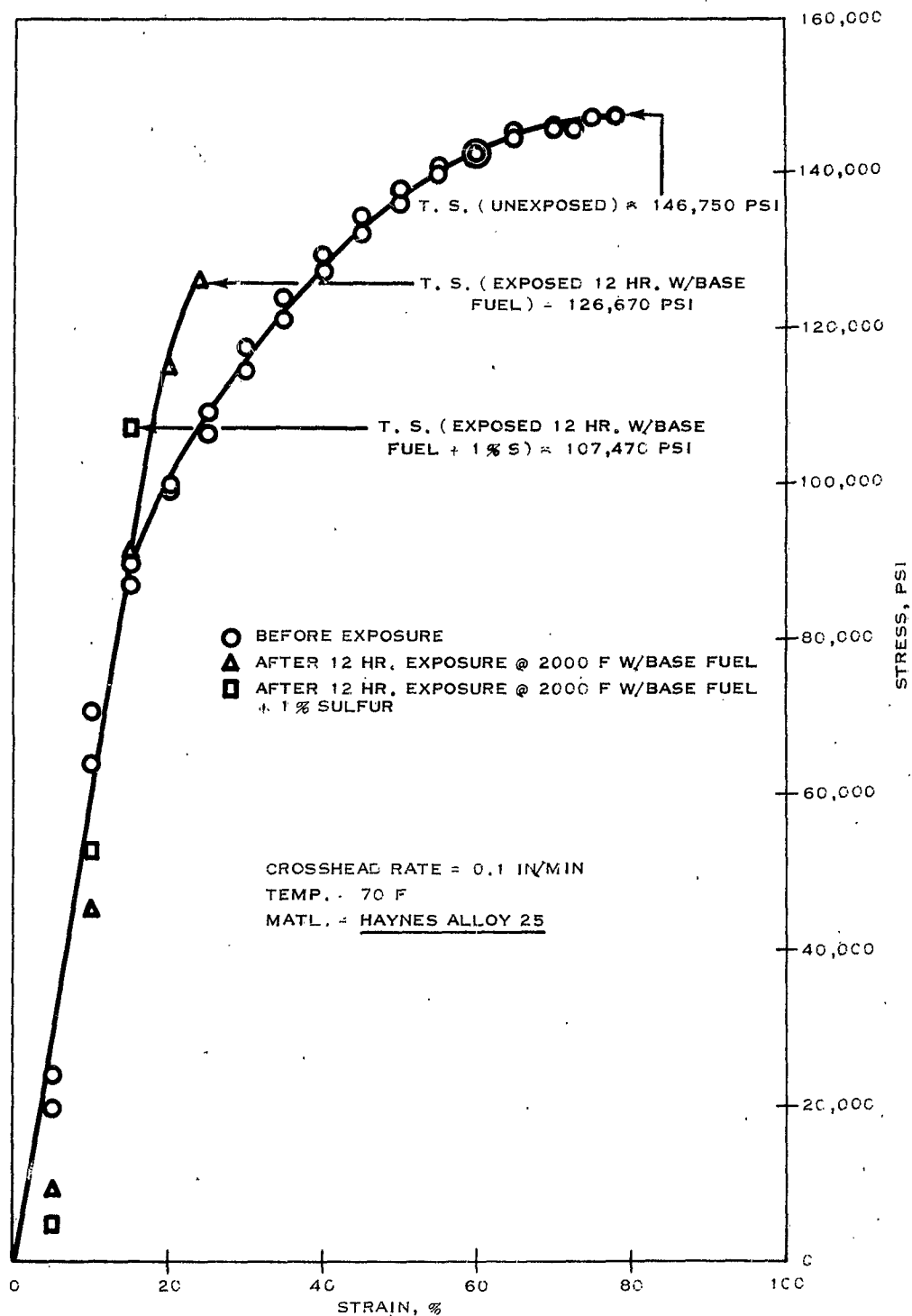


FIGURE 8
 STRESS-STRAIN CURVE FOR HAYNES ALLOY 25 BEFORE AND AFTER
 12 HOURS EXPOSURE TO SULFUR-FREE AND SULFUROUS EXHAUST GAS @ 2000 F
 IN THE PHILLIPS 2-INCH RESEARCH COMBUSTOR

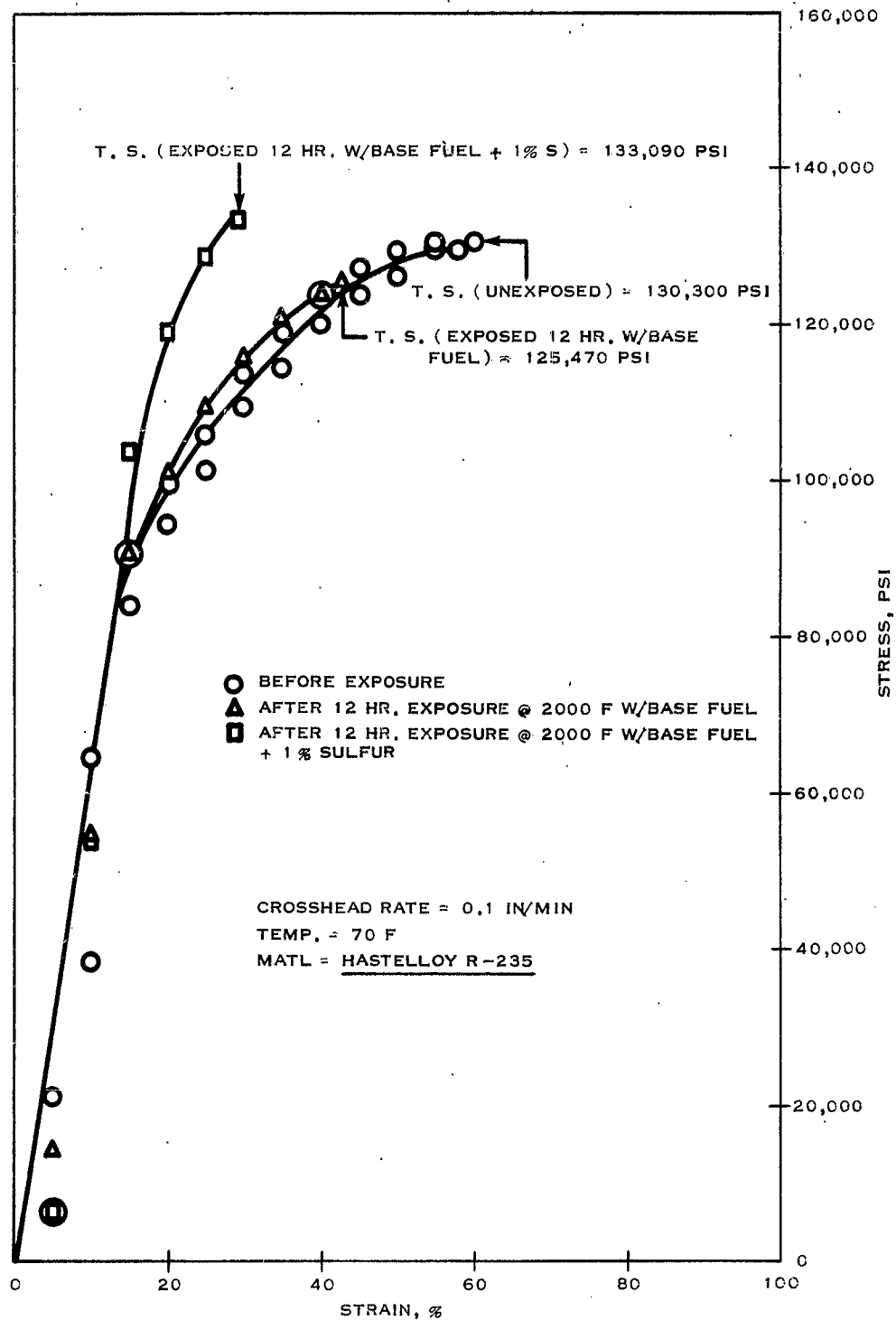


FIGURE 9
STRESS-STRAIN CURVE FOR HASTELLOY R-235 BEFORE AND AFTER 12 HOURS
EXPOSURE TO SULFUR-FREE AND SULFUROUS EXHAUST GAS @ 2000 F
IN THE PHILLIPS 2-INCH RESEARCH COMBUSTOR